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मानक

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Mazdoor Kisan Shakti Sangathan

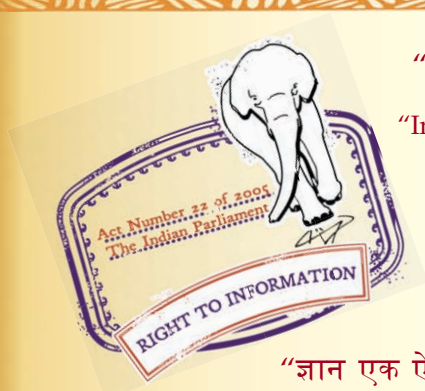
“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

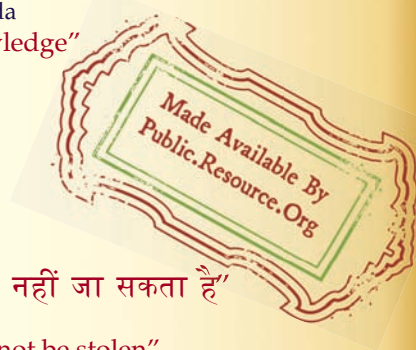
IS 708 (1987): Potassium chlorate for explosive and pyrotechnic industry [CHD 26: Explosives and Pyrotechnics]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**SPECIFICATION FOR
POTASSIUM CHLORATE FOR EXPLOSIVE AND
PYROTECHNIC INDUSTRY**

(Second Revision)

UDC 661.832.3225 : 662.4/5

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

AMENDMENT NO. 1 MARCH 2000
TO
IS 708 : 1987 SPECIFICATION FOR POTASSIUM
CHLORATE FOR EXPLOSIVE AND PYROTECHNIC
INDUSTRY

(Second Revision)

(Page 3, clause A-7.2) — Substitute the following for the existing clause:

'A-7.2 Procedure — Dry the material over sulphuric acid for 24 h. Weigh accurately about 0.3 g of sample and dissolve it in 50 ml of water. Transfer this to a 250 ml volumetric flask and quickly dilute to the mark. Take exactly 50 ml of this solution in a 250 ml conical flask. Add 50 ml of ferrous sulphate (or ferrous ammonium sulphate) solution and about 10 ml of dilute sulphuric acid, close the flask with a stopper to prevent access of air and boil for 15 minutes. Cool the contents of the flask, add 10 ml of manganous sulphate solution and titrate the excess of ferrous sulphate solution against standard potassium permanganate. Run a blank in the same manner with 50 ml of ferrous sulphate (or ferrous ammonium sulphate) solution and about 10 ml of dilute sulphuric acid. The difference between the volume of potassium permanganate consumed in the blank and the sample represents the potassium chlorate content.'

(Page 3, clause A-7.3) — Substitute the following for the existing formula:

$$\text{'Potassium chlorate,} \\ \text{percent by mass} = 2.043 \frac{(V_2 - V_1) \times 5N}{M}$$

(CHD 26)

Indian Standard

SPECIFICATION FOR POTASSIUM CHLORATE FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 30 October 1987, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 Potassium chlorate is an oxidizing agent used in the explosive industry. It is also used in match and pyrotechnic industries.

0.3 This standard was first published in 1956. It was first revised in 1970 in order to include the improved sampling scheme and to align the test methods with those of IS : 5301 - 1969*. The optional defence requirements were also deleted.

0.4 In the present revision, two new requirements, namely, the absence of hypochlorite and the limit for organic matter have been included with a view to facilitate storage and to ensure safety. The title of the standard has also been modified since potassium chlorate is predominantly used in explosive and pyrotechnic industry.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2 - 1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Specification for sodium chlorate.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for potassium chlorate for explosive and pyrotechnic industry.

2. GRADES

2.1 The material shall be of two grades, namely:

- Grade 1* — suitable for use in explosive industry, and
- Grade 2* — suitable for use only in smoke and match compositions.

3. REQUIREMENTS

3.1 Description — The material shall be odourless, in the form of white powder or crystals, and free from grit and visible impurities. The surface of aqueous solution of the material shall not show any oil film.

3.2 Particle Size — 99 percent of the material in the powder form shall pass through 124-micron IS Sieve and 99 percent shall be retained on 90-micron IS Sieve, or as agreed to between the purchaser and the supplier.

3.3 The material shall also comply with the requirements given in Table 1, when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of Table 1.

TABLE 1 REQUIREMENTS FOR POTASSIUM CHLORATE FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

SL No.	CHARACTERISTIC	REQUIREMENT FOR		METHOD OF TEST
		Grade 1	Grade 2 (REF TO CL	No. IN
APPENDIX A)				
(1)	(2)	(3)	(4)	(5)
i)	pH value	5 to 8	5 to 8	A-3
ii)	Moisture, percent by mass, <i>Max</i>	0.05	0.05	A-4
iii)	Insoluble matter, percent by mass, <i>Max</i>	0.05	0.05	A-5
iv)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.03	0.10	A-6
v)	Potassium chlorate (as KClO ₃), percent by mass, <i>Min</i>	99.8	99.25	A-7
vi)	Perchlorates (as KClO ₄), percent by mass, <i>Max</i>	Nil	0.15	A-8
vii)	Sulphates (as SO ₄), percent by mass, <i>Max</i>	0.03	0.03	A-9
viii)	Bromates (as BrO ₃), percent by mass, <i>Max</i>	0.04	0.15	A-10
ix)	Grit	Shall be free from grit		A-11
x)	Nitrogen compounds (as N), percent by mass, <i>Max</i>	0.01	—	A-12
xi)	Alkaline earth and iron, percent by mass, <i>Max</i>	0.02	—	A-13
xii)	Heavy metals	To pass the test	—	A-14
xiii)	Sodium	To pass the test	—	A-15
xiv)	Organic matter, percent by mass, <i>Max</i>	0.01	—	A-16
xv)	Hypochlorite	Shall be free from hypochlorite		A-17

4. PACKING AND MARKING

4.1 Packing — Potassium chlorate shall be packed in containers as agreed to between the purchaser and the supplier and subject to relevant provision of Red Tariff No. 17 of 1954 issued by the Indian Railway Conference Association, with any alterations or additions made thereafter.

4.1.1 Necessary safeguards shall be provided against risk of dangerous explosions which might occur when the material is heated or subjected to concussion or triturated with organic substances, charcoal, sulphur, sulphides, powdered metallic iron, or other oxidizable substances, and precautions shall be taken at all times to prevent accidents by explosions.

4.2 Marking — The package constituting a consignment shall each be legibly marked with a description of the contents, a distinctive lot number, consecutive package number, the tare and net mass, the supplier's initials or recognized trademark, if any, and other relevant provisions of Red Tariff No. 17 of 1954 issued by the Indian Railway

Conference Association, with any alterations or additions made thereafter.

4.2.1 The package may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 The methods for drawing representative samples of the material and criteria for conformity with the requirements of this standard shall be as given in Appendix B.

APPENDIX A (Clause 3.3, and Table 1)

ANALYSIS OF POTASSIUM CHLORATE FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

A-1. GENERAL

A-1.1 Caution — While handling this material, special precautions shall be taken against the risk of dangerous explosions which might occur when the material is heated or subjected to concussion or triturated with organic substances, charcoal, sulphur, sulphides, powdered metallic iron or other oxidizable substances.

A-2. QUALITY OF REAGENTS

A-2.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070 - 1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-3. pH VALUE

A-3.1 Apparatus

A-3.1.1 pH Meter — Use a standard laboratory pH meter (see IS : 2711 - 1979†).

A-3.2 Procedure — Take about 5 g of the material and dissolve it in 100 ml of neutral distilled water. Allow to stand for 5 minutes and then measure the pH of the solution, using a standard pH meter.

A-3.2.1 The pH value of the solution shall be within the limits specified in Table 1.

*Specification for water for general laboratory use (second revision).

†Specification for direct reading pH meters (second revision).

A-4. DETERMINATION OF MOISTURE

A-4.1 Apparatus

A-4.1.1 Weighing Bottle — squat type, with stopper.

A-4.1.2 Electric Oven — maintained at $105 \pm 2^\circ\text{C}$.

A-4.2 Procedure — Weigh accurately about 5 g of the material into a tared, stoppered, weighing bottle. Keep the bottle in an oven maintained at $105 \pm 2^\circ\text{C}$ for 3 hours. Cool in a desiccator and weigh till constant mass.

NOTE — Concentrated sulphuric acid shall not be used as a desiccant.

A-4.3 Calculation

$$\text{Moisture, percent by mass} = 100 \times \frac{M - M_1}{M}$$

where

M = mass in g of the sample before drying, and
 M_1 = mass in g of the sample after drying.

A-5. DETERMINATION OF INSOLUBLE MATTER

A-5.1 Apparatus

A-5.1.1 Filter Crucible — Gooch or sintered glass (G. No. 4).

A-5.2 Procedure — Weigh accurately about 25 g of the material and dissolve in 200 ml of water. Filter through a tared Gooch or sintered glass crucible (G. No. 4). Wash the residue free of all soluble components and dry to constant mass at

105±2°C. Reserve this residue for the test under A-11.

A-5.3 Calculation

Insoluble matter, percent by mass = $100 \times \frac{m}{M}$

where

m = mass in g of the residue, and

M = mass in g of the sample taken for the test.

A-6. TEST FOR CHLORIDES

A-6.1 Apparatus

A-6.1.1 *Nessler Cylinders* — 50-ml capacity.

A-6.2 Reagents

A-6.2.1 *Concentrated Nitric Acid* — See IS : 264 - 1976*.

A-6.2.2 *Silver Nitrate Solution* — approximately 4 percent (m/v).

A-6.2.3 *Standard Chloride Solution* — Dissolve 1.649 g of sodium chloride dried at 105±2°C in water and dilute to 1 000 ml. Dilute 100 ml of this solution to 1 000 ml. One millilitre of this solution contains 0.1 mg of chloride (as Cl).

A-6.3 **Procedure** — Dissolve 1.000 g of Grade 1 material in 30 ml of water, add 5 drops of concentrated nitric acid and 1 ml of silver nitrate solution. In the case of Grade 2 material, dissolve 1.000 g of the material in water and dilute to 100 ml. To 10 ml of this diluted solution, add 5 drops of concentrated nitric acid and 1 ml of silver nitrate solution. Make up the volume to 50 ml mark with water. Carry out a control test, using 3 and 1 ml of standard chloride solution for Grade 1 and Grade 2 material respectively, and add the same quantities of other reagents in the same total volume, of the reaction mixture. Compare the turbidity after 5 minutes.

A-6.3.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-7. DETERMINATION OF POTASSIUM CHLORATE

A-7.1 Reagents

A-7.1.1 *Ferrous Sulphate or Ferrous Ammonium Sulphate Solution* — approximately 0.1 N.

A-7.1.2 *Standard Potassium Permanganate Solution* — 0.1 N.

A-7.1.3 *Manganous Sulphate Solution* — Dissolve 10 g of the material in water and dilute to 100 ml.

A-7.1.4 *Dilute Sulphuric Acid* — approximately 2 N.

A-7.2 **Procedure** — Dry the material over sulphuric acid for 24 hours. Weigh accurately about 0.10 g and dissolve it in 20 ml of water in a 250-ml conical flask. Add exactly 35 ml of ferrous sulphate (or ferrous ammonium sulphate) solution and about 10 ml of dilute sulphuric acid, close the flask with a valve stopper to prevent access of air and boil for 15 minutes. Cool the contents of the flask, add 10 ml of manganous sulphate solution and titrate the excess of ferrous sulphate against standard potassium permanganate. Run a blank in the same manner with 35 ml of ferrous sulphate (or ferrous ammonium sulphate) solution and about 10 ml of dilute sulphuric acid. The difference between the volume of potassium permanganate consumed in the blank and the sample represents the potassium chlorate content.

A-7.3 Calculation

Potassium chlorate,
percent by mass = $2.043 \frac{(V_2 - V_1) N}{M}$

where

V_2 = volume in ml of permanganate solution consumed in the blank experiment,

V_1 = volume in ml of permanganate solution consumed by excess of reducing agent,

N = normality of permanganate solution, and

M = mass in g of the material taken for the test.

A-8. DETERMINATION ON PERCHLORATES

A-8.1 For Grade 1 Material

A-8.1.1 Apparatus

A-8.1.1.1 *Nessler cylinders* — 50-ml capacity.

A-8.1.2 Reagents

A-8.1.2.1 *Methylene blue reagent* — Mix 0.1 ml of 1.6 percent methylene blue solution in water with 25 ml of 50 percent crystallized zinc sulphate solution.

A-8.1.2.2 *Sample potassium chlorate solution* — 10 percent (m/v) solution of Grade 1 material.

A-8.1.2.3 *Chlorate-perchlorate solution* — A solution of 10 percent (m/v) of pure potassium chlorate solution containing 0.01 percent of potassium perchlorate.

A-8.1.2.4 *Potassium nitrate solution* — 40 percent (m/v).

A-8.1.3 **Procedure** — Take 2 ml each of sample potassium chlorate solution and mixed chlorate-perchlorate solution in two Nessler cylinders and add 0.1 ml of potassium nitrate solution and 5 ml of methylene blue reagents and dilute to mark. Compare the colour produced in the two cylinders.

A-8.1.3.1 The material shall be taken as not containing any perchlorate if the colour produced in the cylinder containing the sample potassium chlorate is not darker than in the cylinder containing the chlorate perchlorate solution.

*Specification for nitric acid (second revision).

A-8.2 For Grade 2 Material

A-8.2.1 Apparatus — The apparatus consists of a wide-necked flask (about 300-ml) fitted with a cork carrying a reflux condenser, an inlet tube (6-mm bore) reaching the bottom of the flask, and a short tube (at least 12-mm bore) fitted with a cork.

A-8.2.2 Reagents

A-8.2.2.1 Concentrated hydrochloric acid — See IS : 265 - 1976*.

A-8.2.2.2 Sulphur dioxide — gas.

A-8.2.2.3 Dilute sulphuric acid — approximately 12 N.

A-8.2.2.4 Titanous chloride solution — approximately 3 percent, prepared by diluting 10 ml of 15 percent (m/v) titanous chloride solution to 50 ml with concentrated hydrochloric acid.

A-8.2.2.5 Ammonium thiocyanate solution — approximately 10 percent (m/v).

A-8.2.2.6 Potassium chlorate — free from perchlorates.

A-8.2.2.7 Standard ferric ammonium sulphate solution — approximately 0.1 N, prepared by dissolving 48.22 g of ferric ammonium sulphate in 500 ml of freshly boiled water that has been acidified with 10 ml of dilute sulphuric acid (40 percent by mass). Make up the volume to 1000 ml with freshly boiled water (Solution A).

- a) Measure into a 200-ml conical flask, 25 ml of freshly boiled water, 25 ml of 0.1 N potassium bichromate solution and 15 ml of dilute sulphuric acid (40 percent by mass). Fit the conical flask with a rubber bung bored with two holes, through one of which passes a leading tube, reaching about 50 mm into the flask. Pass a rapid stream of carbon dioxide into the flask for several minutes in order to displace the air. Place the jet of the burette containing the titanous chloride solution in the second hole of the rubber bung and, whilst continuing to pass carbon dioxide, run 20 ml of the titanous chloride solution. Mix the liquid by swirling the flask gently and

titrate the excess of titanous salt with Solution A added from a 25-ml burette, the jet of which is placed inside the second hole in the rubber bung. Towards the end of the titration, that is, when the dark colour due to the titanous salt has nearly disappeared, add 10 ml of ammonium thiocyanate solution and continue the addition of Solution A until a pink colour is obtained that is permanent for one minute. Maintain a slow stream of carbon dioxide throughout the titration.

- b) In the same way, titrate 20 ml of the titanous chloride solution with Solution A from a 50 ml burette but omitting 0.1 N potassium bichromate solution. From the volume of Solution A used in this titration, deduct the volume of ferric ammonium sulphate solution used in the first titration (this difference corresponds to 25 ml of 0.1 N potassium bichromate solution), and calculate the exact normality of ferric ammonium sulphate solution.

A-8.2.3 Procedure — Weigh accurately about 5 g of the material and transfer it completely into the wide-necked flask. Dissolve it in water and pass sulphur dioxide through the solution. Note the progress of the reduction by evolution of heat and the completion of reduction by the solution becoming cold (the time taken is generally 1½ to 2 hours). Remove the excess of sulphur dioxide completely by boiling and by the aid of a current of carbon dioxide passed through the liquid. Complete removal is indicated by the absence of odour of sulphur dioxide as a precaution, continue the boiling gently for 30 minutes after the odour has disappeared. After cooling, add 20 ml of dilute sulphuric acid and dilute the solution with water to 150 ml. Fit the flask to a reflux condenser and pass carbon dioxide through the liquid continuously until the end of the titration. Boil the solution for a few minutes to remove dissolved air. Add an exact volume (say, 10 ml) of titanous chloride solution by means of a pipette through the wide glass tube which is immediately closed with a small cork. Continue refluxing for one hour, after which remove the flask from the condenser and cool in water. Add 5 ml of ammonium thiocyanate solution, and then titrate with Solution A through the glass-lined hole in the cork (Fig. 1).

*Specification for hydrochloric acid (second revision).

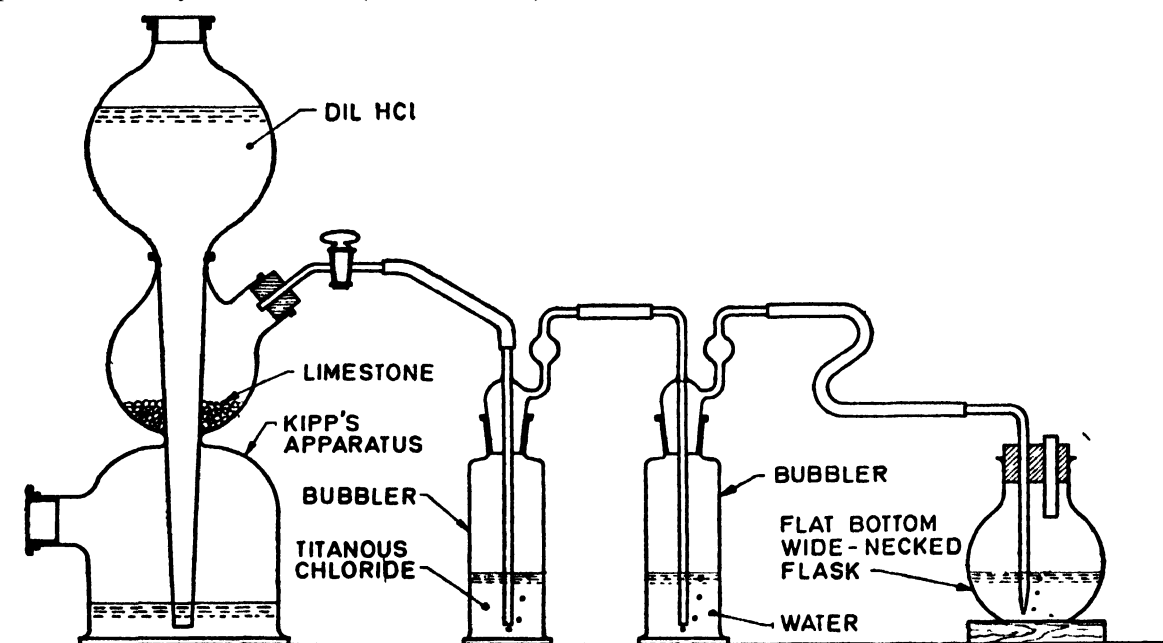


FIG. 1 APPARATUS FOR TITRATION IN AN INERT (CARBON DIOXIDE) ATMOSPHERE

A-8.2.3.1 Carry out a blank determination using potassium chlorate (perchlorate-free) and other reagents used in the test under **A-8.2.3**.

A-8.2.3.2 Apparatus for titration in inert (carbon dioxide) atmosphere and for preservation and addition of titanous chloride solution are shown in Fig. 1 and 2 respectively.

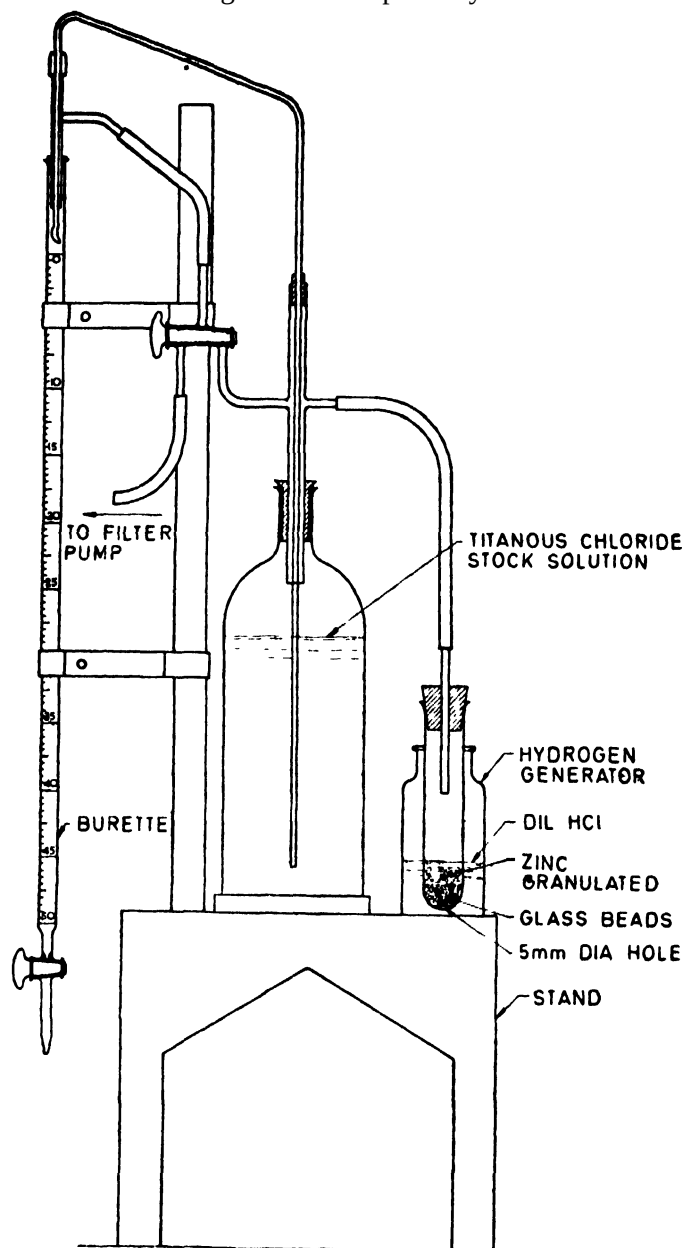


FIG. 2 APPARATUS FOR PRESERVATION AND ADDITION OF TITANOUS CHLORIDE SOLUTION

A-8.2.4 Calculation

Potassium perchlorate

$$\left(\text{as } \text{KClO}_4 \right), \text{ percent by mass} = 1.73 \times \frac{(V_1 - V_2) N}{M}$$

where

V_1 = volume in ml of standard ferric ammonium sulphate solution required for the blank determination (see **A-8.2.3.1**),

V_2 = volume in ml of standard ferric ammonium sulphate solution (see **A-8.2.3**) required for test with the material,

N = normality of standard ferric ammonium sulphate solution, and

M = mass in g of the material taken for the test.

A-9. TEST FOR SULPHATES

A-9.1 Apparatus

A-9.1.1 Nessler Cylinders — 50-ml capacity.

A-9.2 Reagents

A-9.2.1 Dilute Hydrochloric Acid — 1 N.

A-9.2.2 Barium Chloride Solution — approximately 10 percent (m/v).

A-9.2.3 Standard Sulphate Solution — Dissolve 0.148 g of ignited sodium sulphate (Na_2SO_4) in water and dilute to 1000 ml. One millilitre of this solution contains 0.1 mg of sulphate (SO_4).

A-9.3 Procedure — Dissolve 1 g of the material in 20 ml of water, add 5 ml of dilute hydrochloric acid and 2 ml of barium chloride solution. Transfer to a Nessler cylinder and dilute to mark. Carry out a control test in another Nessler cylinder using 3 ml of standard sulphite solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture.

A-9.3.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that in the control test.

A-10. DETERMINATION OF BROMATES

A-10.1 Reagents

A-10.1.1 Potassium Iodide Solution — approximately 10 percent (m/v).

A-10.1.2 Dilute Hydrochloric Acid — approximately 1 N.

A-10.1.3 Standard Sodium Thiosulphate Solution — 0.1 N freshly prepared.

A-10.1.4 Starch Solution — Triturate 5 g of pure starch and 0.01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into one litre of boiling water, boil for 3 minutes, allow the solution to cool, and decant off the clear liquid. The solution used for the test shall be freshly prepared.

A-10.1.5 Potassium Chlorate — bromate-free.

A-10.2 Procedure — Weigh accurately about 2 g of the material into a thoroughly cleaned iodine flask and dissolve in about 100 ml of water. Add 5 ml of potassium iodide solution and 5 ml of dilute hydrochloric acid. Stopper the flask and allow to stand in dark for 45 minutes. At the end of this period, add a little of starch solution and titrate the liberated iodine with freshly prepared standard thiosulphate solution.

A-10.2.1 Carry out a blank determination using potassium chlorate (bromate-free) and other reagents used in the test under **A-10.2**.

A-10.3 Calculation

$$\text{Bromate (as BrO}_3\text{), percent by mass} = 2.13 \times \frac{(V_1 - V_2)N}{M}$$

where

V_1 = volume in ml of standard sodium thiosulphate solution required for the test with the material,

V_2 = volume in ml of standard sodium thiosulphate solution required for the blank determination,

N = normality of standard sodium thiosulphate solution, and

M = mass in g of the material taken for the test.

A-11. DETERMINATION OF GRIT

A-11.1 Procedure — Brush the residue, as obtained under A-5.1, into a beaker, treat with a few ml of aqua regia [3 volumes of concentrated hydrochloric acid (conforming to IS : 265-1976*) and 1 volume of concentrated nitric acid (conforming to IS : 264-1976†)] and then warm gently; cool, dilute the solution and filter through a filter paper. Wash the residue on the filter paper, ignite, and then weigh. Brush the ignited residue on 63-micron IS Sieve and weigh the material retained.

A-12. TEST FOR NITROGEN COMPOUNDS**A-12.1 Apparatus**

A-12.1.1 Nessler Cylinders — 50-ml capacity.

A-12.2 Reagents

A-12.2.1 Sodium Hydroxide Solution — 10 percent.

A-12.2.2 Devarda's Alloy — containing 45 parts aluminium, 50 parts copper and 5 parts zinc.

A-12.2.3 Nessler Solution — Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly while stirring, saturated mercuric chloride solution until a slight permanent precipitate is formed. Add 30 g of potassium hydroxide and when it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

A-12.2.4 Standard Nitrogen Solution — Dissolve 0.3819 g of ammonium chloride in enough water to make up the volume to 1000 ml. One millilitre of this contains 0.1 mg of nitrogen (as N).

A-12.3 Procedure — Dissolve 1.000 g of the material in 40 ml of water. Add 1 g of powdered Devarda's alloy and 10 ml of 10 percent sodium hydroxide solution. Allow to stand for 2 hours protected from loss or access to ammonia, then distil 40 ml, collecting the distillate in 5 ml

of water containing 1 drop of dilute hydrochloric acid. Transfer the distillate to a Nessler cylinder, and add 1 ml of 10 percent sodium hydroxide solution and 2 ml of Nessler solution. Carry out the control tests in exactly the same manner using 1 ml of nitrogen solution for Grade 1 and 5 ml for Grade 2.

A-12.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control tests.

A-13. DETERMINATION OF ALKALINE EARTHS AND IRON**A-13.1 Reagents**

A-13.1.1 Ammonium Oxalate — Dissolve 3.5 g of ammonium oxalate in water to make 100 ml.

A-13.1.2 Ammonium Phosphate Solution — Dissolve 13 g of diammonium hydrogen phosphate $[(\text{NH}_4)_2 \text{HPO}_4]$ in water to make 100 ml.

A-13.1.3 Ammonium Hydroxide Solution — sp gr 0.90.

A-13.2 Procedure — Dissolve 10.0 g of the material in 200 ml of hot water and filter. Add to the filtrate, 5 ml of ammonium oxalate solution, 3 ml of ammonium phosphate solution and 30 ml of ammonium hydroxide solution. Allow the solution to stand overnight. If there is any precipitate, filter, wash well with hot water, ignite and weigh.

A-13.3 Calculation — Calculate the mass of the precipitate as percentage by mass of the material taken for the test.

A-14. TEST FOR HEAVY METALS**A-14.1 Apparatus**

A-14.1.1 Nessler Cylinders — 50-ml capacity.

A-14.2 Reagents

A-14.2.1 Hydrochloric Acid — See IS : 265-1976*.

A-14.2.2 Dilute Acetic Acid — approximately 1 N.

A-14.2.3 Hydrogen Sulphide Solution — saturated solution in water.

A-14.3 Procedure — Dissolve 2.0 g of the material in 5 ml of water and 5 ml of hydrochloric acid. Evaporate the solution to dryness on a steam-bath. Repeat the evaporation with 2 ml of dilute acetic acid and then dilute with water to 30 ml. Transfer the solution to the Nessler cylinder, add 10 ml of hydrogen sulphide solution and mix well.

A-14.3.1 The material shall be regarded to have passed the test if no appreciable darkening of colour is observed after 5 minutes.

*Specification for hydrochloric acid (second revision).

†Specification for nitric acid (second revision).

*Specification for hydrochloric acid (second revision).

A-15. TEST FOR SODIUM

A-15.1 Procedure — Dip a clean platinum wire (fused at the end of a glass rod) into a 10 percent (m/v) solution of the material in water and hold it in the colourless Bunsen flame.

A-15.1.1 The material shall be taken to have satisfied the requirement of the test if no pronounced yellow colour, such as to mask the characteristic violet colour of potassium, is produced.

A-16. TEST FOR ORGANIC MATTER**A-16.1 Apparatus**

A-16.1.1 Silica Crucible — of porosity G 4.

A-16.2 Reagents

A-16.2.1 Hydrochloric Acid — See IS : 265-1976*.

A-16.2.2 Silver Nitrate

A-16.3 Procedure — Weigh about 25 g of sample and dissolve in 300 ml of hot distilled water. Heat the solution on a water bath for 1 h. Filter the solution through a previously weighed silica crucible. Wash with hot water. Treat the residue with hydrochloric acid several times, altogether with 10 ml. Wash the residue with boiling hot distilled water until it gives a negative test for chloride on

reaction with silver nitrate. Dry it at 105-110°C under constant pressure. Weigh the residue and then calcinate it under constant pressure and weigh it again.

A-16.4 Calculation

$$\text{Organic matter, percent by mass} = \frac{(G_1 - G_2)}{G} \times 100$$

where

G_1 — mass in g of the dried residue treated with hydrochloric acid.

G_2 — mass in g of the calcinated residue, and

G = mass in g of potassium chlorate taken.

A-17. DETERMINATION OF HYPOCHLORITE CONTENT**A-17.1 Reagents**

A-17.1.1 Potassium Iodide — 10 percent solution.

A-17.1.2 Starch — 1 percent solution.

A-17.2 Procedure — Weigh out 5 g of sample and dissolve it in 100 ml of water. Add 5 ml of potassium iodide solution and 2.3 ml of starch solution. The sample shall be considered to be free from hypochlorite if no change is observed in potassium iodide within 30 minutes.

*Specification for hydrochloric acid (second revision).

APPENDIX B

(Clause 5.1)

SAMPLING OF POTASSIUM CHLORATE, TECHNICAL**B-1. GENERAL REQUIREMENTS OF SAMPLING**

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Necessary safeguards as prescribed in 4.1.1 shall be provided in taking out the material from the containers for sampling.

B-1.2 The samples and the sampling implement shall be well protected from any possible contamination.

B-1.3 The sampled material shall be placed in suitable, dry, clean container which shall be marked with all relevant details after filling.

B-1.4 Samples shall be taken from different parts of the selected containers with the help of suitable sampling implements.

B-2. SCALE OF SAMPLING

B-2.1 All the containers in a single consignment of potassium chlorate of the same grade and belonging to the same batch of manufacture shall be grouped together to constitute a lot.

B-2.1.1 Samples shall be used for each lot separately for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be selected from each lot depends on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE	No. OF CONTAINERS TO BE SELECTED FOR SAMPLING
N	n
(1)	(2)
Up to 25	3
26 to 50	4
51 to 100	5
101 and above	7

B-2.3 These containers shall be selected at random from the lot and in order to ensure the randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

'Starting from any container, count them in one order as 1, 2, 3, ..., up to r and so on, where r is an integral part of N/n (N and n being the lot size and the sample size, respectively). Every r th

container thus counted shall be withdrawn to constitute the sample.

B-3. NUMBER OF TESTS

B-3.1 From each of the selected containers, sufficient quantity of potassium chlorate shall be withdrawn to conduct individual sieve analysis for particle size in case the material is in powder form. From the portions collected for sieve analysis, about 100 g of potassium chlorate shall be transferred to sample containers and these filled containers are termed individual samples.

B-3.2 Out of the remaining portions from each selected container, equal amount of material shall

be withdrawn and mixed together to form a composite sample weighing 150 g.

B-3.3 Tests for particle size and potassium chlorate content shall be conducted for each of the selected containers in the sample. Tests for all the other characteristics shall be made on the composite sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 The lot shall be accepted as conforming to this specification if all the test results, as mentioned in **B-3.3**, satisfy the corresponding requirements given in Table 1.

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